Application of superabsorbent polymers (SAP) in cementitious materials with blended cements

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Internal curing agent in concrete and mortars

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This paper is part of the larger study on the application of superabsorbent polymers (SAP) in concrete construction. The focus here is placed on the assessment of SAP efficiency as an internal curing agent in cementitious materials with blended cements. The paper presents the outcomes of two experimental studies on polymer modified mortars containing ground granulated blast-furnace slag (GGBS) and fly ash (FA). Results show that SAP has a positive influence on mortars with blended cements due to its capacity to release water for longer periods. Any SAP addition will result in significant reductions in autogenous shrinkage. Long term compressive strength values of SAP modified mortars are comparable with the reference specimens despite an initial drop due to polymer collapse. The use of SAPs in blended cements meets the challenges of the construction industry to produce new composites with improved performance.

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This paper forms the second part of a series on superabsorbent polymers (SAP) as a novel admixture for concrete and mortars. In the first part, an overview of SAP application in ordinary cementitious composites has been presented [1]. This article, in turn, addresses the effect of SAP on cementitious materials with blended cements, in particular with ground granulated blast-furnace slag (GGBS) and fly ash (FA).

Blended cements comprise Portland cement (PC) and supplementary cementitious materials (SCM) which exhibit certain hydraulic and/or pozzolanic activities. The use of SCM conserves energy and has a positive environmental impact [2]-[3]. Since the process of cement fabrication requires very high temperatures (usually above 1400 °C), it involves the release of considerable amount of carbon dioxide into the atmosphere, consumes large amounts of non-renewable raw materials, and generates harmful pollutants, such as dioxins and heavy metals [3]. Therefore, the replacement of Portland cement with SCM leads to a more environmentally sustainable concrete industry [2]-[8].The use of GGBS and FA prevents these industrial by-products from penetrating the soil, contributes to the cost reduction of building materials, and provides solution to the environmental issues associated with waste management [5]. SCM can also enhance the properties of concrete, such as strength and durability [2][6], and hence can increase their popularity in the construction industry. Figure 1 shows SEM micrographs of PC, GGBS and FA.

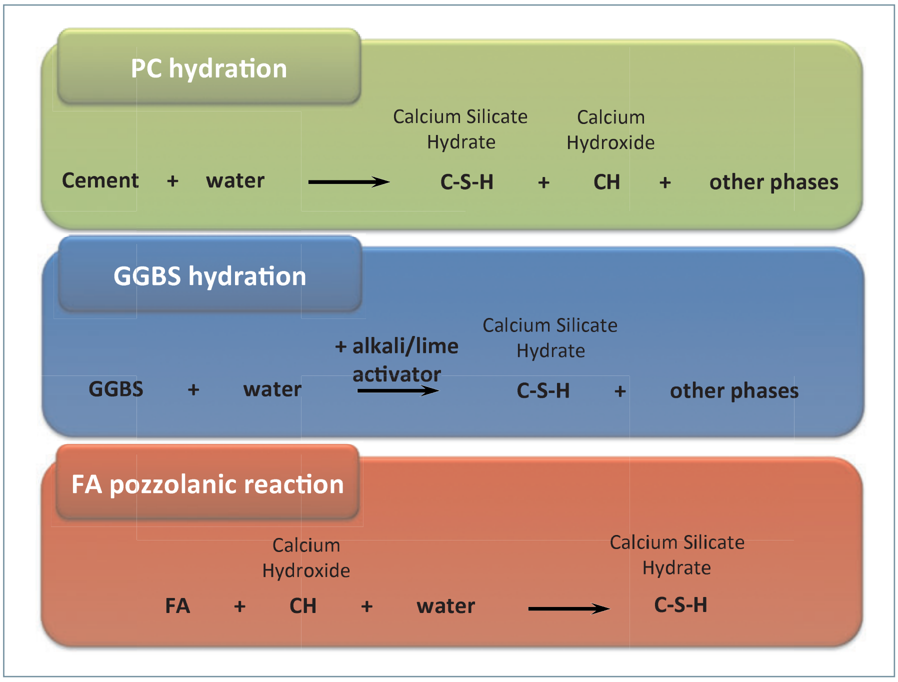

Fig. 2: PC, GGBS and FA reactions.

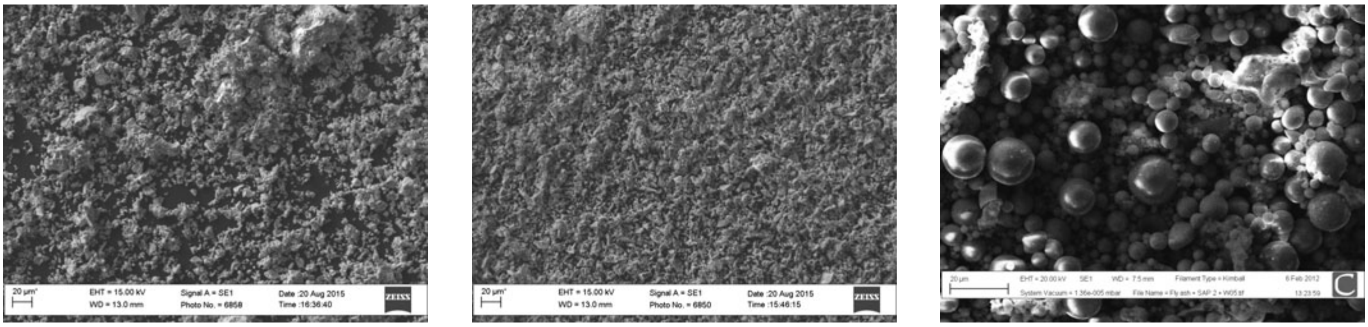

Fig. 1: SEM micrographs of PC (CEM I), GGBS and FA, respectively.

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To be considered an SCM, a by-product has to exhibit at least one of the three following properties: (i) hydraulic properties, i.e., the ability to harden by reactions with water (e.g. PC, GGBS), which may show, for example, by a pH increase (alkaline activation); (ii) pozzolanic properties, i.e., the ability to harden by a reaction with lime in an aqueous medium (e.g. FA, blast-furnace slag, silica fume), where lime is usually provided by PC; and (iii) activating properties, i.e., the ability to promote the hydration of pozzolanic/hydraulic materials by providing lime (e.g. lime rich waste) and/or initiating/accelerating their hydration (alkaline and sulphate activation) [8]. Figure 2 shows three equations corresponding to PC hydration, GGBS hydration and pozzolanic reaction of FA.

GGBS is a by-product of the steel industry [2][9]. Slag is a latent hydraulic material whose reactivity depends on its chemical composition, fineness, glass content, temperature, and alkali concentration of the reacting system. In the process of slag hydration, which is activated by alkalis/lime present in PC, calcium silicate hydrate (CSH) is formed (Figure 2). The use of GGBS in cement and concrete results in low hydration heat, increased setting times, increased strength at later ages, reduced permeability, and high resistance to chloride penetration, sulphate attack and alkali-silica reaction [9][10].

On the other hand, FA (a pozzolanic material also known as pulverised fuel ash) is a waste product from coal-fired power plants [2][11]. During the pozzolanic reaction, silicic acid ($Si(OH)_4$, present in FA) will react with calcium hydroxide (CH, from cement hydration) in the presence of water and forms CSH gel (Figure 2). FA may also contain aluminate phases ($Al(OH)_4^-$) that, in combination with silica, react with CH to form calcium-alumino-silicate hydrates (i.e. C_2ASH_8). The pozzolanic activity of FA depends upon its fineness, calcium content, structure, specific surface, particle size distribution and loss on ignition (LOI) content [2]. Partial replacement of PC by FA may lead to lower early strengths, higher ultimate strengths,

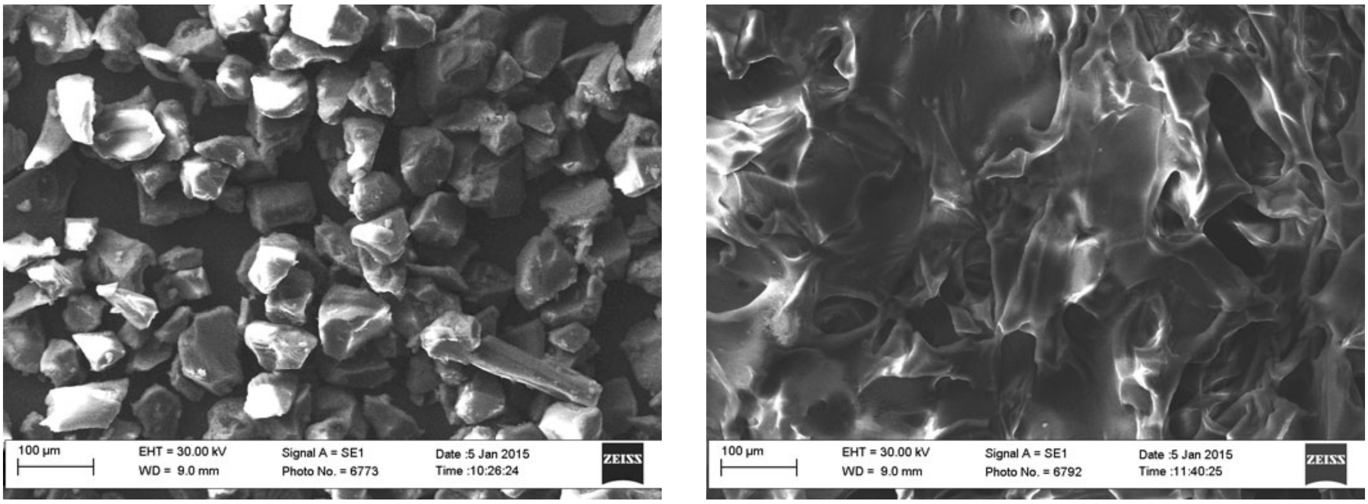

Fig. 3: SEM micrographs of SAP in dry and wet conditions, respectively.

lower heat of hydration, lower permeability, greater resistance to lime leaching, greater resistance to sulphate and sea water expansion, and inhibition of the alkali-silica expansion [12].

Addition of GGBS or FA in concrete will lead to setting retardation and strength increment in the long term. Both SCMs may slow down cementitious reactions, meaning that more time is necessary to complete the formation of hydrated products. Consequently, the curing process is longer and its control should be more effective, provided that a sufficient amount of water is available. Superabsorbent polymers (SAP) can aid the hydration process by acting as an internal curing agent. Their high capacity to absorb water from fresh mixes and release it in either fresh or hardened states can lead to an improvement of some properties of concrete, such as a reduction in autogenous shrinkage [1][13][14]. Figure 3 shows SEM micrographs of SAP used in the experimental study in dry and wet conditions, respectively.

Hence, this paper aims at presenting the experimental results of two studies with different types of SAPs in mortars with blended cements (containing GGBS and FA). Three types of SAPs have been considered in the experimental programme: SAP X, SAP Y and SAP Z, with water absorption capacities in cement paste solution of 25-30 g/g, 35 g/g and 10 g/g, respectively. The effects of SAP have been assessed in terms of autogenous shrinkage (AS) by the corrugated tubes method [15]) and mechanical properties [16]. Table 1 shows compositions of mortars used in this study. The water/cement (w/c) ratio of FA mortars was adjusted to maintain the same worka-

Tab 1: Mortar specimen composition

Specimen	Type of SCM	Content of SCM	Type of SAP	Cement/sand (c/s)	Water/cement (w/c)
RS-25	GGBS	25%	–	1:2	0,50
XS-25	GGBS	25%	X	1:2	0,50
YS-25	GGBS	25%	Y	1:2	0,50
RS-50	GGBS	50%	–	1:2	0,50
XS-50	GGBS	50%	X	1:2	0,50
YS-50	GGBS	50%	Y	1:2	0,50
RA-11	FA	25%	–	1:1	0,45
XA-11	FA	25%	X	1:1	0,45
ZA-11	FA	25%	Z	1:1	0,45
RA-12	FA	25%	–	1:2	0,45
XA-12	FA	25%	X	1:2	0,45
ZA-12	FA	25%	Z	1:2	0,45

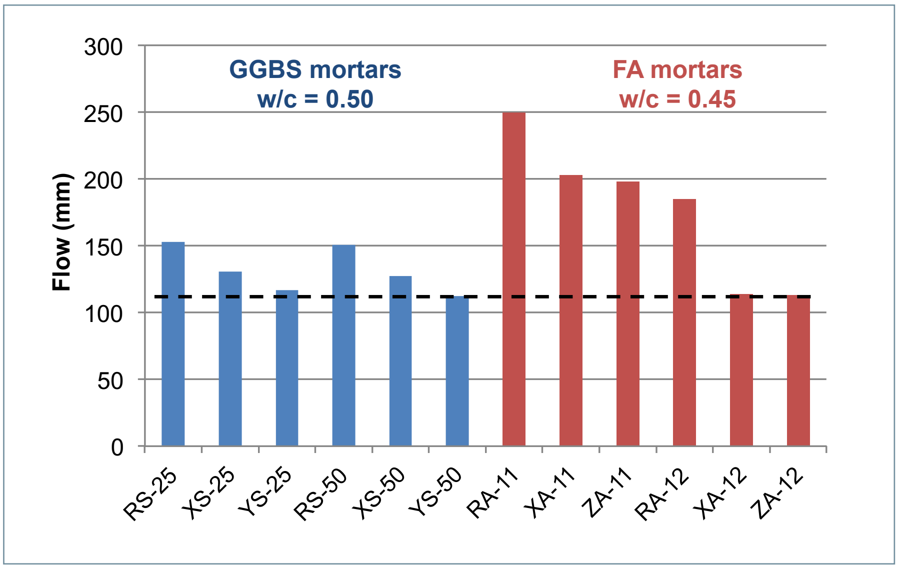

Fig. 4: Results of flow testing, dashed line indicating the same level of consistency for w/c ratio determination.

bility level of GGBS mortar [17]; the driest mortar with slag (considering 50% of GGBS and SAP Y) used as a standard to determine the consistency of the driest mortar with FA considering SAP Z and proportion 1:2 (Figure 4). Thereafter, the adjusted w/c ratio was retained for all mortars with FA.

Ground granulated blast-furnace slag (GGBS)

The effect of two types of SAP (X and Y) on mortars featuring GGBS has been evaluated by considering two levels of PC replacement by slag (25% and 50%) (Table 1). Respective specimens without GGBS have been presented in the previous paper [1]. In general, concrete made with GGBS presents higher autogenous shrinkage (AS) than the reference concrete without GGBS [10][18][19]. This is generally attributed to higher chemical shrinkage, presence of finer pores, removal of calcium hydroxide (CH) as a shrinkage restraint, and a reduction in pore humidity associated with hydration reactions [20].

Increments in AS can be related to the higher hydration degree of GGBS, and therefore, greater degree of self-desiccation. In addition, CSH formed during the reaction (Figure 2) produces a chemical shrinkage, since the volume of the hydrated products is less than the sum of volumes of water and initial anhydrous products [18]. Thus, greater chemical shrinkage of concrete containing GGBS will lead to faster and greater self-desiccation and result in larger AS [10][19].

Moreover, the addition of slag in cementitious matrices can lead to a denser structure caused by the formation of smaller pores. Finer pores, in turn, contribute to a lower internal relative humidity which may induce higher capillary forces during self-desiccation processes increasing AS [10][21]. This approach can be understood assuming a linear elastic behaviour of matrices with slag [22]. On the other hand, autogenous shrinkage may also be caused by plastic deformation and the creep of cement matrices under internal stress caused by the conversion of ettringite into monosulphate and consumption of CH [22]. Slag may have a minimal pozzolanic activity on account of its combination with CH released by the hydration of PC in a similar way as FA reactions (Figure 2). The consumption of CH crystals may remove internal restraints in concrete and allow the occurrence of further shrinkage.

This behaviour of increasing AS can be reduced by incrementing the GGBS content in the mix [18]-[20]. The higher the level of PC replacement by GGBS, the greater self-desiccation shrinkage will be. In fact, this can be verified by experimental results as shown in Figure 5. Mortars with 25% and 50% of GGBS have presented AS at 42 days of about 500 and 600 µm/m, respectively.

However, this behaviour is dramatically altered by the addition of SAP. While mortars with GGBS exhibit AS greater than 500 µm/m, their equivalent specimens with SAPs will reach values below 120 µm/m, (over 75% reduction). It seems then that the

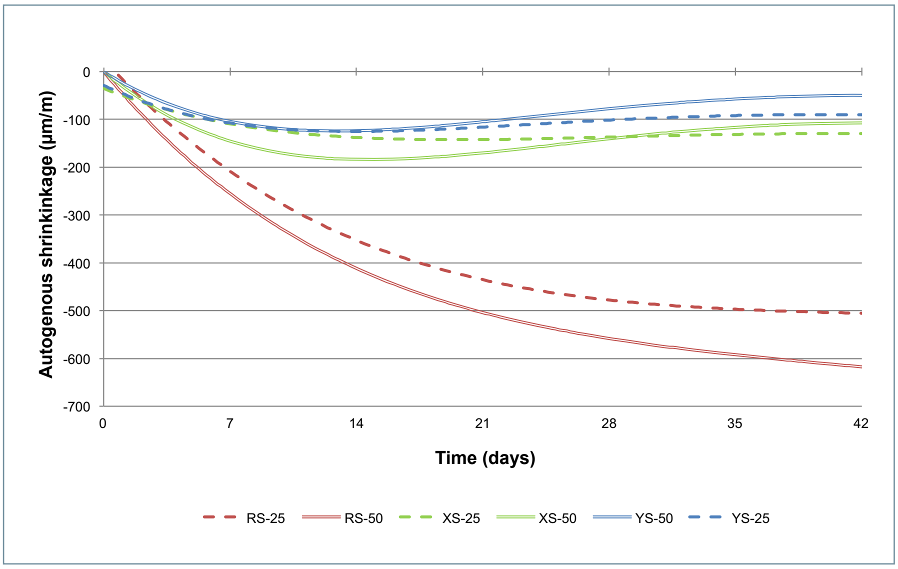

Fig. 5: Autogenous shrinkage for mortars with ground granulated blast furnace slag (GGBS).

greater water absorption capacity of SAP, the greater AS reduction will turn out to be. SAP may act as a "water-bubble bag" functioning as a water provider and tension releaser. SAP can be considered a small water storage distributed across the mix providing a sort of water reservoir for longer hydration reactions [23][24]. In particular in the case of mortars featuring GGBS, this longer water supply can contribute to more efficient reactions of slag and hence can mitigate self-desiccation in early ages.

On the other hand, SAP can also perform as a tension releaser. Mortars with GGBS tend to have a finer pore structure which leads to an increase of tensile stresses provoked by water menisci between the pore walls [18]. Since SAP increases air content in fresh mortars [1], these additional air voids can release capillary tension. In consequence, forces generated during the self-desiccation process can be decreased and their effect on AS is minimised. In this way, incrementing the air content by SAP will counteract and improve the effect of finer pore structures provided by the presence of GGBS.

As regarding the mechanical characteristics of cementitious materials with slag, there is a consensus that GGBS decreases strength in early age but increases it in the long term [2][9][19][25]. This is due to the fact that the hydraulic capacity of slag tends to be slow in its appearance since it needs to be activated to start hydration processes and the formation of CSH [18]. Moreover, the surface area and particle size distribution of slag are strongly related to the strength of mortar featuring GGBS [9]. Basically, the hydration of slag may be accelerated by increasing its specific surface, using chemical activators (alkalis in PC) or raising reaction temperature [18]. In this experimental study, PC was finer than GGBS with finenesses of 410 m^2/kg and 390 m^2/kg, respectively. This may explain the decreases in compressive and flexural strength for higher GGBS contents (as illustrated in Figures 6 and 7). In addition, the level of alkalis available for the activation of slag hydration in the mix decreases with the replacement of PC by GGBS. Even in the presence of SAP, this pattern of reduced compressive strength in mortars with higher GGBS contents is maintained.

Moreover, SAP appears to have no influence or, if any, merely a small reduction in compressive strength in mortars with GGBS, especially at early ages. However, this loss in strength seems to be partly recovered with time, resulting from ongoing hydration facilitated by the internal curing mechanisms [24][26][27]. In fact, Figure 6 shows that, despite a modest reduction in compressive strength for mortars with SAP Y in the first days, their values have been in the same order than the reference specimens at 90 days. SAP promotes creation of a dense network of CSH in a collapsed state, and hence leading to pore closures and increased compressive strength [28].

As reported previously by [26], SAP may have a positive effect on the tensile characteristics of GGBS mortars, especially at later ages. However, experimental results presented here do not support this statement; only specimens with SAP Y and 25% of GGBS have outperformed their respective reference mortars (Figure 7). In this way, the effect of different types of SAP appears to be more relevant on flexural properties of mortars with slag. SAP performance in GGBS mortars can be influenced by its capacity and kinetics sorption, and concentration of pore solution given by different slag contents in the mix [1][24][28]. Overall, the difference of flexural strength by the increment of GGBS has been decreased at later ages. As this property is more sensitive to smaller pores, later hydration products of slag can gradually fill those finer voids. This effect results in tensile strength values of the same order when mortars with different GGBS contents are compared in advanced ages.

Therefore, SAP has a positive effect on mitigating AS in mortars with GGBS. This reduction is more pronounced for higher contents of GGBS. While compressive strength is not significantly affected by SAP addition at later ages, flexural strength seems to be more sensitive to the type of SAP and amount of GGBS in the mix.

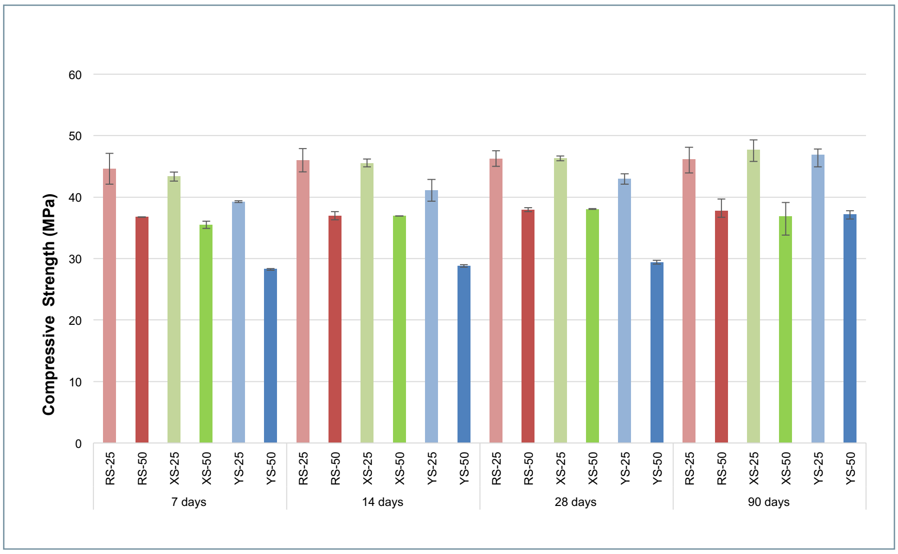

Fig. 6: Compressive strength results for mortars featuring GGBS.

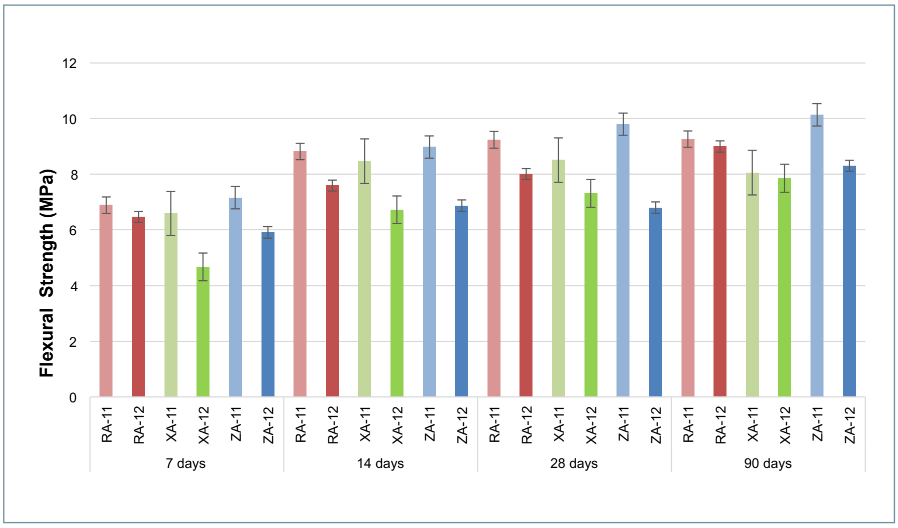

Fig. 7: Flexural strength results for mortars featuring GGBS.

Fly ash (FA)

As shown in Figure 8, the effect of SAP modification on autogenous shrinkage of mortars with FA was clearly noticeable. Values have been highest for the reference mixes with a cement-to-sand ratio (c/s) of 1:1 from the very beginning. Different SAPs have not affected the results to the same extent. The most noticeable effect has been recorded for specimens with SAP Z and c/s = 1:1 where AS was almost four times smaller than for the reference specimens. SAP X has rendered a three-time reduction after seven days after specimen preparation. Between the first and the third week, AS hardly changed. In the third week, the swelling of specimens containing SAP X and SAP Z started to cease. Even though a lower AS for reference specimens with c/s=1:2 (RA-12) was recorded, its extent was identical at later ages to reference specimens with a lower amount of sand (RA-11). A similar trend for all SAP specimens was observed: a small swelling was recorded between the second and the fifth week with no further linear changes observed later.

Since AS for SAP Z diminished significantly during the first week and increased for the reference specimen, it is likely that the majority of stored water was used up during this period. It can therefore be assumed that SAP Z provided water to the cementitious matrix as soon as self-desiccation occurred. However, a small swelling up to the fifth week may indicate that water desorption by SAP Z lasted at least up to this time. SAP X proved to have a positive effect on AS reduction comparable to of SAP Z.

The type of polymer, and in consequence its water absorption capacity and absorption/desorption kinetics, were the most influential factors on the AS progress. Despite a similar effect on AS, it seems that the absorption/desorption behaviour of SAP X and SAP Z was very dissimilar. The substantial reduction of workability by SAP Z was an indication of a very quick rate of water absorption. Nonetheless, the noticeable limitation in AS during the first week may suggest a reduction of self-desiccation by a substantial water supply on account of collapsing SAP Z. In turn, a rather insignificant effect of SAP X on workability and delay in autogenous shrinkage cease by approximately a week by comparison to SAP Z could be recorded. Furthermore, FA may play a significant role on the described behaviour. Several researchers [29]-[31] have suggested that AS increases along with an increase in the degree of fly ash hydration. Berry et al. [31] reported that the amount of ettringite in FA cement mixes was higher than in mixes with CEM I in the first 5 hours after mixing, leading to increased water consumption. As a result, empty voids might appear and consequently AS will increase. However, contrary results have also been reported [32].

Specimens with a cement-to sand-ratio of 1:2 have displayed a slightly lower compressive strength than specimens with a 1:1 ratio, as illustrated in Figure 9. A similar mechanical performance of concretes with Portland cement and concretes with 20-30% was reported in several studies [33-37]. Overall, a comparable pattern was detected for the reference specimens and those modified by SAPs. Compressive strength results have increased for all specimens at later ages (after four weeks). Flexural strength values for all specimens have shown some increase during the first two weeks, then stabilised for the next two weeks, and thereafter increased further (Figure 10). However, no significant alterations were observed after four weeks of curing for specimens with a c/s ratio of 1:2. Increases in strength values for specimens with c/s = 1:1 cement to sand were recorded after the fourth week with the most pronounced ones for the reference specimens.

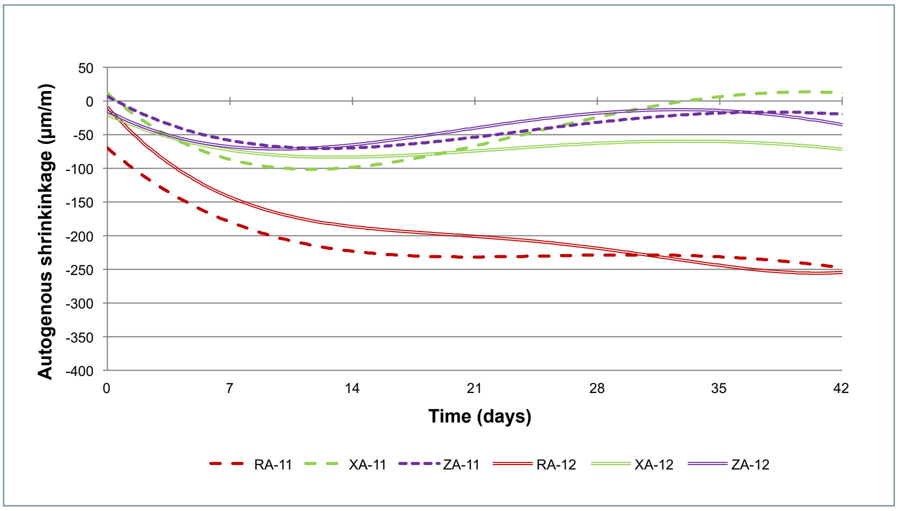

Fig. 8: Autogenous shrinkage for mortars featuring FA

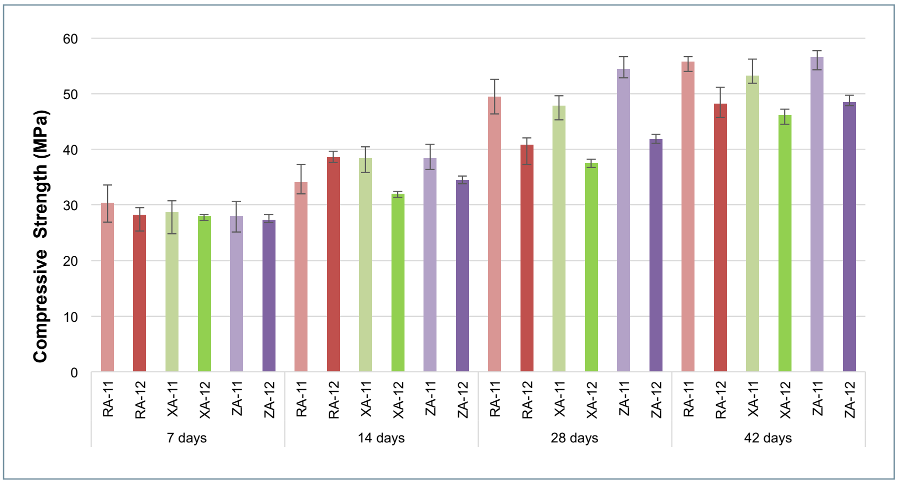

Fig. 9: Compressive strength results for mortars featuring FA.

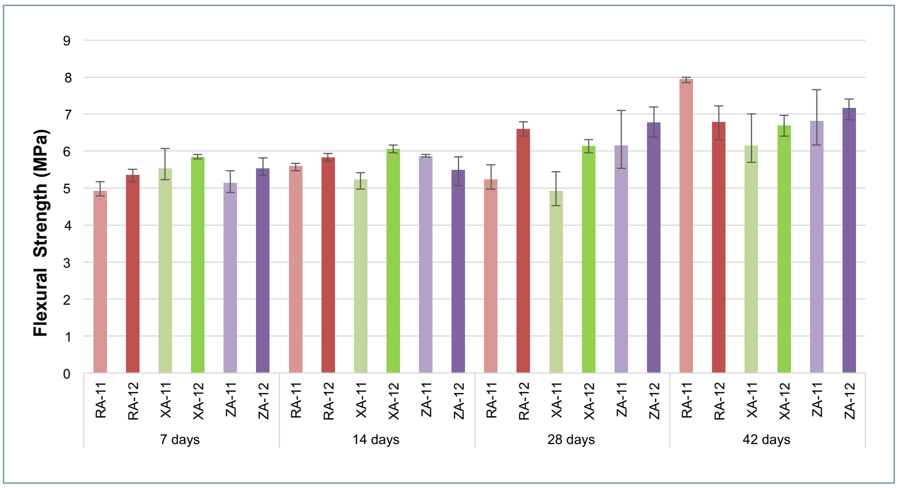

Fig. 10: Flexural strength results for mortars featuring FA.

Unlike for specimens with c/s=1:1, the effect of SAPs on the performance of mortars with c/s= 1:2 is rather negligible. Small but steady increases can be observed for both compressive and flexural strength for all tested specimens. This can be associated with the significant role of higher aggregate content. Due to the lower cement paste, the content formation of 'SAP pores' (i.e. pores remaining after a collapse of SAP) only has a limited effect on strength development [28][38][39]. 'SAP pores' are sufficiently small and do not adversely affect compressive strength. On the other hand, the occurrence of 'SAP pores' influences flexural strength, which is sensitive to porosity changes in all ranges of pore size.

Furthermore, an FA addition to Portland cement has a notable influence on the hydration process and microstructure development in cementitious composites regardless of any modifications by SAP.

FA reactivity level has been attributed to its particle size distribution [40][41]. Such reactivity was found to be directly proportional to the amount of particles below 10 µm, and inversely proportional to particles bigger than 45 µm [40]. Moreover, higher amounts of aggregates in the matrix do not significantly influence the density of CSH gel in the interfacial transition zone in composites with FA cements. Since the sizes of fly ash particles are mostly below 40 µm, they act as effective fillers for voids in the vicinity of aggregates and play a dominant role in ensuring the packing of cement particles ("wall effect"). It is also believed that even after 91 days of hydration, parts of fly ash still remain unreacted [40][42]. These unreacted particles, exhibiting a higher modulus of elasticity than the matrix, may act as micro-aggregates in cement pastes and could be considered a composite material on a micro-scale.

Final remarks and outlook

Blended cements play an increasingly important role in the construction industry due to their environmental benefits and the generally improved performance of cementitious materials. However, their longer hydration process caused by pozzolanic reactions make them vulnerable to self-desiccation and in consequence to internal cracking. The application of superabsorbent polymers (SAP) can solve this problem by providing a gradual supply of water for hydration.

Experimental investigations have confirmed that SAP additions in both GGBS and FA mortars result in a considerable reduction of autogenous shrinkage. A slight swelling can even be noticed in both materials after the second week due to an enhanced formation of hydration products.

Mechanical properties are influenced not only by the type of SAP (chemical composition, cross-linking and water absorption capacity) but on the cement type. The concentration of ions in a pore solution (due to different SCM used) plays a critical role in absorption and desorption processes. However, a similar trend can be observed for specimens after 6 weeks of curing. Despite initial drops in strength due to a collapse of SAP, the compressive strength values for SAP-modified mortars are comparable with their respective reference specimens.

Nevertheless, it must be stated that further studies are essential before any practical recommendations can be made, especially when different combinations of supplementary cementitious materials are used.

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